PATENT COOPERATION TREATY

From th INTERNATIONAL BUREAU

PCT

NOTIFICATION OF THE RECORDING OF A CHANGE

(PCT Rule 92bis.1 and Administrative Instructions, Section 422)

To:

PHILLIPS & LEIGH
7 Staple Inn
Holborn
London WC1V 7QF
ROYAUME-UNI

Date of mailing (day/month/year)
07 December 1998 (07.12.98)

Applicant's or agent's file reference
FP-08-0466

IMPORTANT NOTIFICATION

International application No.
PCT/GB97/01667

International filing date (day/month/year)
20 June 1997 (20.06.97)

1. The following indications appeared on record concerning:

[X] the applicant [X] the inventor [ ] the agent [ ] the common representative

Name and Address
JUBB, Gary, Anthony
11 Lawnswood House
Church Avenue
Stourport-on-Severn
Worcestershire DY13 9OX
United Kingdom

State of Nationality: GB
State of Residence: GB
Telephone No.:
Facsimile No.:
Teleprinter No.:

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

[ ] the person [ ] the name [X] the address [ ] the nationality [ ] the residence

Name and Address
JUBB, Gary, Anthony
62 Dunlin Drive
Kidderminster
Worcestershire DY10 4TA
United Kingdom

State of Nationality: GB
State of Residence: GB
Telephone No.:
Facsimile No.:
Teleprinter No.:

3. Further observations, if necessary:

4. A copy of this notification has been sent to:

[X] the receiving Office
[ ] the International Searching Authority
[ ] the International Preliminary Examining Authority
[ ] the designated Offices concerned
[X] the elected Offices concerned
[ ] other:

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland
Facsimile No.: (41-22) 740.14.35

Authorized officer
Ting Zhao
Telephone No.: (41-22) 338.83.38

PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

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(PCT Rule 92bis.1 and Administrative Instructions, Section 422)

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International application No.
PCT/GB97/01667

International filing date (day/month/year)
20 June 1997 (20.06.97)

1. The following indications appeared on record concerning:

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Name and Address
LOWE, Alison, Jane
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Ferndale Estate
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United Kingdom

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State of Residenc: GB
Telephone No.:
Facsimile No.:
Teleprinter No.:

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

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Kidderminster
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[ ] the International Preliminary Examining Authority
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[X] the elected Offices concerned
[ ] other:

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1211 Geneva 20, Switzerland
Facsimile No.: (41-22) 740.14.35

Authorized officer
Ting Zhao
Telephone No.: (41-22) 338.83.38

PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

To:

United States Patent and Trademark Office
(Box PCT)
Crystal Plaza 2
Washington, DC 20231
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 30 January 1998 (30.01.98)	
International application No. PCT/GB97/01667	Applicant's or agent's file reference FP-08-0466
International filing date (day/month/year) 20 June 1997 (20.06.97)	Priority date (day/month/year) 21 June 1996 (21.06.96)
Applicant JUBB, Gary, Anthony et al	

1. The designated Office is hereby notified of its election made:

[X] in the demand filed with the International Preliminary Examining Authority on:

09 January 1998 (09.01.98)

[ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] was

[ ] was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer J. Leitao
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

Form PCT/IB/331 (July 1992)

1863879

PATENT COOPERATION TREA

PCT

NOTIFICATION OF THE RECORDING OF A CHANGE

(PCT Rule 92bis.1 and Administrative Instructions, Section 422)

From the INTERNATIONAL BUREAU

To:

PHILLIPS & LEIGH
7 Staple Inn
Holborn
London WC1V 7QF
ROYAUME-UNI

Date of mailing (day/month/year)
07 December 1998 (07.12.98)

Applicant's or agent's file reference
FP-08-0466

IMPORTANT NOTIFICATION

International application No.
PCT/GB97/01667

International filing date (day/month/year)
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United Kingdom

State of Nationality: GB
State of Residence: GB
Telephone No.:
Facsimile No.:
Teleprinter No.:

3. Further observations, if necessary:

4. A copy of this notification has been sent to:

[X] the receiving Office
[ ] the International Searching Authority
[ ] the International Preliminary Examining Authority
[ ] the designated Offices concerned
[X] the elected Offices concerned
[ ] other:

The International Bureau of WIPO
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1211 Geneva 20, Switzerland

Facsimile No.: (41-22) 740.14.35

Authorized officer
Ting Zhao

Telephone No.: (41-22) 338.83.38

Form PCT/IB/306 (March 1994)

002376982

Copy for the Elected Office (EO/US)

PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF THE RECORDING OF A CHANGE

(PCT Rule 92bis.1 and Administrative Instructions, Section 422)

To:

PHILLIPS & LEIGH
7 Staple Inn
Holborn
London WC1V 7QF
ROYAUME-UNI

Date of mailing (day/month/year)
07 December 1998 (07.12.98)

Applicant's or agent's file reference
FP-08-0466

IMPORTANT NOTIFICATION

International application No.
PCT/GB97/01667

International filing date (day/month/year)
20 June 1997 (20.06.97)

1. The following indications appeared on record concerning:

[X] the applicant [X] the inventor [ ] the agent [ ] the common representative

Name and Address
LOWE, Alison, Jane
11 Mayfield Close
Ferndale Estate
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United Kingdom

State of Nationality: GB
State of Residence: GB
Telephone No.:
Facsimile No.:
Teleprinter No.:

3. Further observations, if necessary:

4. A copy of this notification has been sent to:

[X] the receiving Office
[ ] the International Searching Authority
[ ] the International Preliminary Examining Authority
[ ] the designated Offices concerned
[X] the elected Offices concerned
[ ] other:

The International Bureau of WIPO
34, chemin des Colombettes
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Facsimile No.: (41-22) 740.14.35

Authorized officer
Ting Zhao

Telephone No.: (41-22) 338.83.38

002376986

09/202,730

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference	FOR FURTHER ACTION	see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.
FP-08-0466		
International application No.	International filing date (day/month/year)	(Earliest) Priority Date (day/month/year)
PCT/GB 97/01667	20/06/1997	21/06/1996

Applicant

THE MORGAN CRUCIBLE COMPANY PLC et al.

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

[X] It is also accompanied by a copy of each prior art document cited in this report.

1. [ ] **Certain claims were found unsearchable** (see Box I).

2. [ ] **Unity of invention is lacking** (see Box II).

3. [ ] The international application contains disclosure of a **nucleotide and/or amino acid sequence listing** and the international search was carried out on the basis of the sequence listing
   - [ ] filed with the international application.
   - [ ] furnished by the applicant separately from the international application,
     - [ ] but not accompanied by a statement to the effect that it did not include matter going beyond the disclosure in the international application as filed.
   - [ ] Transcribed by this Authority

4. With regard to the **title**,
   - [X] the text is approved as submitted by the applicant.
   - [ ] the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,
   - [X] the text is approved as submitted by the applicant.
   - [ ] the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this International Search Report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is:
   Figure No. --
   - [ ] as suggested by the applicant.
   - [ ] because the applicant failed to suggest a figure.
   - [ ] because this figure better characterizes the invention.
   - [ ] None of the figures.

PATENT COOPERATION TREAT

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:
PHILLIPS & LEIGH
7 Staple Inn
Holborn
London WC1V 7QF
GRANDE BRETAGNE

NOTIFICATION OF TRANSMITTAL OF INTERNATIONAL PRELIMINARY EXAMINATION REPORT

JTB

(PCT Rule 71.1)

Date of mailing *(day/month/year)* 18.09.98

Applicant's or agent's file reference
FP-08-0466

IMPORTANT NOTIFICATION

International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/GB 97/01667	20/06/1997	21/06/1996

Applicant
THE MORGAN CRUCIBLE COMPANY PLC et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.

2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.

3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. **REMINDER**

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices)(Article 39(1))(see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Nam and mailing address of the IPEA

European Patent Office, P.B. 5818 Patentlaan 2
NL-2280 HV Rijswijk - Netherlands
Tel.: (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer
M. Dekker
Tel.: 4046

Telephone No.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
FP-08-0466		
International application No.	International filing date (*day/month/year*)	Priority date (*day/month/year*)
PCT/GB 97/ 01667	20/06/1997	21/06/1996
International Patent Classification (IPC) or national classification and IPC		
C03C13/00		
Applicant		
THE MORGAN CRUCIBLE COMPANY PLC et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This **REPORT** consists of a total of 5 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consists of a total of 5 sheets.

3. This report contains indications and corresponding pages relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
   - VI ☐ Certain documents cited
   - VII ☐ Certain defects in the international application
   - VIII ☒ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
09/01/1998	18.09.98

Nam and mailing address of the IPEA

European Patent Office, P.B. 5818 Patentlaan 2
NL-2280 HV Rijswijk - Netherlands
Tel.: (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

van Bomm I, L.

Telephone No. 02241

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/GB97/01667

I. Basis of the report

1. This report has been drawn up on the basis of *(Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.)*

☐ the international application as originally filed

☒ the description, pages 3 - 8 , as originally filed
pages , filed with the demand
pages 1, 2 , filed with the letter of 03.04.98

☒ the claims, Nos. , as originally filed
Nos. , as amended under Article 19
Nos. , filed with the demand
Nos. 1 - 7 , filed with the letter of 03.04.98

☐ the drawings, sheets / fig. , as originally filed
sheets / fig. , filed with the demand
sheets / fig. , filed with the letter of

2. The amendments have resulted in the cancellation of:

☐ the description, pages:

☐ the claims, Nos.

☐ the drawings, sheets / fig.

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2 (c)).

4. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty	Claims	1 - 7	YES
	Claims		NO
Inventive Step	Claims	1 - 7	YES
	Claims		NO
Industrial Applicability	Claims	1 - 7	YES
	Claims		NO

2. Citations and Explanations

i. Reference is made to the following documents:
D1: WO- A- 93 15028
D2: WO- A- 95 29135
D3: WO- A- 93 22251
D4: WO- A- 89 12032
D5: DE- A- 44 17 230

ii. The claims of the application define the use of B_2O_3 and/or P_2O_5 for improving the refractoriness of inorganic fibres, the fibres comprising SiO_2 and CaO and optionally MgO, and having a shrinkage of less than 3.5% when exposed to 800°C and/or 1000°C for 24 hours. The claims also define saline soluble inorganic fibres having a shrinkage of less than 3.5% when exposed to 800°C and/or 1000°C for 24 hours, the fibres comprising SiO_2 and CaO and optionally MgO, and either or both of B_2O_3 and P_2O_5.

iii. D1 describes saline soluble inorganic fibres consisting essentially of SiO_2, CaO and MgO and having low shrinkage at 800°C and 1000°C.
The subject- matter of the claims differs from D1 in that the fibres comprise either or both of B_2O_3 and P_2O_5.

D2 - D5 all describe inorganic fibres comprising SiO_2, CaO, MgO and either or both of B_2O_3 and P_2O_5.
The subject- matter of the claims differs from D2 - D5 in that the fibres have a shrinkage of less than 3.5% when exposed to 800°C and/or 1000°C for 24 hours.

Therefore, novelty w.r.t. D1 - D5 is acknowledged for all claims.

iv. The problem to be solved in D1 is to improve the refractoriness in that sense that a wider window of compositions can be used for fibers with a desired low shrinkage and high saline solubility.
The problem is solved by adding either or both of B2O3 and P2O5 in certain amounts to fiber compositions with certain amounts of SiO2, CaO and MgO.
The addition of either or both of B2O3 and P2O5 to fiber compositions is known from D2 - D5.
However, it was not obvious to combine the teaching of D2 - D5 with D1, because
1. the addition of either or both of B2O3 and P2O5 in D2 - D5 was for a different reason (for improving saline solubility, and not for improving refractoriness), and
2. In D1 it is explicitly stated that impurities such as B2O3 are undesirable, if a certain resistance to temperature is to be achieved.
Therefore, inventive step is acknowledged.

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

The feature in claims 1, 5, 6 and 7 of the fibers "having a shrinkage of less than 3.5% when exposed to 800°C for 24 hours and having a shrinkage of less than 3.5% when exposed to 1000°C for 24 hours" is considered clear for the following reasons:
Said feature is not considered as a "result to be achieved", but as a product parameter which in itself is clear, distinctive and readily measurable.
The features mentioned on page 3, paragraphs 1 - 4, are not seen as restrictive, but as information enabling the skilled person to manufacture fibers that fulfill the shrinkage requirement.

SALINE SOLUBLE INORGANIC FIBRES

This invention relates to saline soluble inorganic fibres.

Saline soluble inorganic fibres have been described in several patent specifications, see for example WO93/15028. Fibres are required to be soluble in saline solution so that inhaled or ingested fibres dissolve rather than providing a source of irritation or otherwise affecting health. WO93/15028 showed that fibres comprising SiO_2, CaO and MgO and having a silica content of greater than 58% (or greater than 58% plus 0.5 times (wt%MgO - 10) if MgO > 10wt%) had suitable shrinkage characteristics at 800°C and 1000°C to be usable as refractory materials. A further feature of WO93/15028 was the use of the percentage of non-bridging oxygens present to predict the solubility of fibres in physiological saline solution.

Various subsequent applications have described the effect of P_2O_5 and B_2O_3 on solubility - see for example WO95/29135. P_2O_5 is alleged to have a solubilising effect on such fibres. WO93/22251 refers to use of P_2O_5 and Na_2O to improve solubility of fibres. WO89/12032 and DE 4417230 disclose fibres containing SiO_2, CaO, MgO, and B_2O_3.

The German government have proposed a fibre classification which turns on a variable K_I which is defined as:

$$K_I = \Sigma(\text{Na,K,B,Ca,Mg,Ba -oxide}) - 2^* \text{ Al-oxide}$$

(the amounts of the oxides being expressed as weight %)

According to the proposed fibre classification if K_I is greater than 40 the fibre requires no health warnings. If K_I lies between 30 and 40 the fibre requires health warnings to be made. If K_I is less than 30 more serious marking is required (it is labelled as a carcinogen). It is readily apparent that it is difficult to provide a high K_I fibre (K_I>40) while still providing a refractory fibre like that of WO93/15028 (SiO_2>58wt%), there being a very narrow window of compositions to meet.

As a result of investigating fibre compositions that may meet the fibre classification and yet still be refractory enough to meet the standard of WO93/15028 (shrinkage of less than 3.5% at both 800°C and1000°C) the applicants have found that addition of P_2O_5 to compositions allows a broader range of refractory fibres to be produced than had previously been appreciated.

They have also found that B_2O_3, previously thought to be extremely detrimental to refractoriness, has a similar, although lesser, effect and that both P_2O_5 and B_2O_3 may be used in the fibres of WO93/15028.

The applicants have found that the refractoriness of the P_2O_5 and B_2O_3 containing fibres of the present invention is dependent on the sum of the amounts of SiO_2 and P_2O_5 (expressed in wt%)

It appears that a further factor that may be important in determining the refractoriness of a fibre is the percentage of non-bridging oxygens. If this percentage is 61.4% or more (calculated on the basis of the amounts of the components SiO_2, CaO, MgO, P_2O_5, and B_2O_3) the fibres tend to fail shrinkage tests at 800°C and 1000°C (failure being defined as a shrinkage of 3.5% or more).

The scope of the invention is apparent from the claims in the light of the following description.

The percentage of non-bridging oxygens (%N.B.O.) is calculated by converting the weight percentages of SiO_2, CaO, MgO, P_2O_5, and B_2O_3 to molar amounts and inserting these amounts into the equation:-

$$\%N.B.O. = \frac{2*(CaO + MgO + P_2O_5 + B_2O_3)}{(2*SiO_2 + CaO + MgO + 5 \times P_2O_5 + 3 \times B_2O_3)} \times 100$$

The reason the amounts of CaO, MgO, P_2O_5, and B_2O_3 are doubled in the numerator to this equation is that each contributes two non-bridging oxygens. The reason terms are multiplied in the denominator to this equation is to reflect the number of oxygen atoms each molecular formula possesses.

Table I shows the results of a first set of shrinkage and solubility tests on compositions comprising SiO_2, CaO, MgO, P_2O_5, and B_2O_3 as main

CLAIMS

1. The use of either or both P_2O_5 and B_2O_3 as a component to improve the refractoriness of inorganic fibres comprising SiO_2, and CaO and/or MgO, to produce inorganic fibres having a composition having a shrinkage of less than 3.5% when exposed to 1000°C for 24 hours and having a shrinkage of less than 3.5% when exposed to 800°C for 24 hours, the fibres having a composition:-

SiO_2	44wt% or more
CaO	20 – 40wt%
MgO	0 - 18wt%
P_2O_5	0- 12.5wt%
B_2O_3	0 - 4wt%

and in which

$SiO_2 + P_2O_5 - (58 + (\text{if MgO} > 10,\ 0.5 \times (\text{MgO} - 10) \text{ else } 0)) > -2.4\text{wt\%}$

2. The use of either or both P_2O_5 and B_2O_3 as a component to improve the refractoriness of inorganic fibres as claimed in claim 1 in which the percentage of non-bridging oxygens is less than 61.4%.

3. The use of either or both P_2O_5 and B_2O_3 as a component to improve the refractoriness of inorganic fibres as claimed in claim 1 in which the fibres fall within the compositional range:-

SiO_2	52 - <58wt% [52 - <58+0.5×(MgO-10)wt% if MgO > 10wt%]
CaO	22 - 40wt%
MgO	0 - 17.5wt%
MgO + CaO	< 42wt%
P_2O_5	0.5 - 10wt%
B_2O_3	0 - 2wt%

4. The use of either or both P_2O_5 and B_2O_3 as a component to improve the refractoriness of inorganic fibres in which the fibres fall within the compositional range:-

SiO_2	44.34 - 62.48
CaO	20.36 - 39.4wt%
MgO	0.62 - 21.16wt%
P_2O_5	0 – 12.01wt%
B_2O_3	0 - 3.54wt%

and in which

$SiO_2 + P_2O_5 - (58 + (\text{if MgO} > 10,\ 0.5 \times (\text{MgO} - 10) \text{ else } 0)) > -2.4\text{wt\%}$

5. Saline soluble inorganic fibres having a shrinkage of less than 3.5% when exposed to 1000°C for 24 hours and having a shrinkage of less than 3.5% when exposed to 800°C for 24 hours, in which:-

$SiO_2 + P_2O_5 - (58 + (\text{if MgO} > 10,\ 0.5 \times (\text{MgO} - 10) \text{ else } 0)) > -2.4\text{wt\%}$

and comprising:-

SiO_2	52 - <58wt% [52 - <58+0.5'(MgO-10)wt% if MgO > 10wt%]
CaO	22 - 40wt%
MgO	0 - 17.5wt%
MgO + CaO	< 42wt%
P_2O_5	0.5 - 10wt%
B_2O_3	0 - 2wt%

and in which the percentage of non-bridging oxygens calculated on the basis of the amounts of the above named components is less than 61.4%.

6. Saline soluble inorganic fibres having a shrinkage of less than 3.5% when exposed to 1000°C for 24 hours and having a shrinkage of less than 3.5% when exposed to 800°C for 24 hours, in which:-

$SiO_2 + P_2O_5 - (58 + (\text{if MgO} > 10,\ 0.5 \times (\text{MgO} - 10) \text{ else } 0)) > -2.4\text{wt\%}$

and comprising:-

SiO_2	44.34 - 62.48
CaO	20.36 - 39.4wt%
MgO	0.62 - 21.16wt%

and also comprising either or both of:-

P_2O_5	0 – 12.01wt%
B_2O_3	0 - 3.54wt%

7. Saline soluble inorganic fibres having a shrinkage of less than 3.5% when exposed to 1000°C for 24 hours and having a shrinkage of less than 3.5% when exposed to 800°C for 24 hours, in which:-

$SiO_2 + P_2O_5 - (58 + (\text{if MgO} > 10,\ 0.5 \times (\text{MgO} - 10) \text{ else } 0)) > -2.4\text{wt\%}$

and comprising:-

SiO_2	52.4 - 57.85wt%
CaO	22.2 - 39.4wt%
MgO	1.96 - 17.4wt%
P_2O_5	0.82 - 7.8wt%
B_2O_3	0 - 1.95wt%
Al_2O_3	<1wt%

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Published
With international search report.

(54) Title: SALINE SOLUBLE INORGANIC FIBRES

(57) Abstract

The use of P_2O_5 and/or B_2O_3 as a component to improve the refractoriness of inorganic fibres comprising SiO_2, and CaO and/or MgO is described. The inorganic fibres have a composition such that $SiO_2 + P_2O_5$-(58 + (if MgO > 10, 0.5 x (MgO-10) else 0)) > -2.4 wt.%.

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 97/01667

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C03C13/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C03C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 93 22251 A (SAINT GOBAIN ISOVER ;HOLSTEIN WOLFGANG (DE); LOHE PETER (DE); SCHW) 11 November 1993 see page 1, line 37 - page 3, line 36; example 6 ---	1-3,5-7, 9
X	WO 89 12032 A (MANVILLE SALES CORP) 14 December 1989 see page 9, paragraph 3 - page 10, paragraph 2; examples 164,166-170 ---	1-3,6,7
X	DE 44 17 230 A (GRUENZWEIG & HARTMANN) 23 November 1995 see example 2 --- -/--	1,2,6

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

1

Date of the actual completion of the international search	Date of mailing of the international search report
22 September 1997	30.09.97

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax (+31-70) 340-3016	Van Bommel, L

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 93 15028 A (MORGAN CRUCIBLE CO) 5 August 1993 cited in the application see page 7, paragraph 4 - page 9, paragraph 3; examples	6,7
X	WO 92 09536 A (PAROC OY AB) 11 June 1992 see example C	6
A	WO 96 01793 A (ROCKWOOL AB ;PERANDER MICHAEL (FI); ROENNLOEF BJOERN (FI)) 25 January 1996 see page 5, line 28 - page 7, line 10	1-10
A	WO 95 29135 A (ROCKWOOL INT ;JENSEN SOREN LUND (DK); CHRISTENSEN VERMUND RUST (DK) 2 November 1995 cited in the application see page 3, line 26 - page 7, line 26	1-10

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/GB 97/01667

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INTERNATIONAL SEARCH REPORT

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International Application No
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PATENT COOPERATION TREATY

PCT

21 SEP 1998

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
FP-08-0466		
International application No.	International filing date (day/month/year)	Priority date (day/month/year)
PCT/GB 97/ 01667	20/06/1997	21/06/1996
International Patent Classification (IPC) or national classification and IPC		
C03C13/00		
Applicant		
THE MORGAN CRUCIBLE COMPANY PLC et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 5 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consists of a total of 5 sheets.

3. This report contains indications and corresponding pages relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
   - VI ☐ Certain documents cited
   - VII ☐ Certain defects in the international application
   - VIII ☒ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
09/01/1998	18.09.98
Name and mailing address of the IPEA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Netherlands Tel.: (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	van Bommel, L. Telephone No. 02241

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/GB97/01667

I. Basis of th report

1. This report has been drawn up on the basis of *(Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.)*

☐ the international application as originally filed

☒ the description, pages 3 - 8 , as originally filed
pages , filed with the demand
pages 1, 2 , filed with the letter of 03.04.98

☒ the claims, Nos. , as originally filed
Nos. , as amended under Article 19
Nos. , filed with the demand
Nos. 1 - 7 , filed with the letter of 03.04.98

☐ the drawings, sheets / fig. , as originally filed
sheets / fig. , filed with the demand
sheets / fig. , filed with the letter of

2. The amendments have resulted in the cancellation of:

☐ the description, pages:

☐ the claims, Nos.

☐ the drawings, sheets / fig.

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2 (c)).

4. Additional observations, if necessary:

V. **Reasoned statement under Articl 35(2) with regard to novelty, inventive st p or industrial applicability; citations and explanations supporting such statement**

1. Statement

Novelty	Claims	1 - 7	YES
	Claims		NO
Inventive Step	Claims	1 - 7	YES
	Claims		NO
Industrial Applicability	Claims	1 - 7	YES
	Claims		NO

2. Citations and Explanations

i. Reference is made to the following documents:
D1: WO- A- 93 15028
D2: WO- A- 95 29135
D3: WO- A- 93 22251
D4: WO- A- 89 12032
D5: DE- A- 44 17 230

ii. The claims of the application define the use of B_2O_3 and/or P_2O_5 for improving the refractoriness of inorganic fibres, the fibres comprising SiO_2 and CaO and optionally MgO, and having a shrinkage of less than 3.5% when exposed to 800°C and/or 1000°C for 24 hours. The claims also define saline soluble inorganic fibres having a shrinkage of less than 3.5% when exposed to 800°C and/or 1000°C for 24 hours, the fibres comprising SiO_2 and CaO and optionally MgO, and either or both of B_2O_3 and P_2O_5.

iii. D1 describes saline soluble inorganic fibres consisting essentially of SiO_2, CaO and MgO and having low shrinkage at 800°C and 1000°C.
The subject- matter of the claims differs from D1 in that the fibres comprise either or both of B_2O_3 and P_2O_5.

D2 - D5 all describe inorganic fibres comprising SiO_2, CaO, MgO and either or both of B_2O_3 and P_2O_5.
The subject- matter of the claims differs from D2 - D5 in that the fibres have a shrinkage of less than 3.5% when exposed to 800°C and/or 1000°C for 24 hours.

Therefore, novelty w.r.t. D1 - D5 is acknowledged for all claims.

iv. The problem to be solved in D1 is to improve the refractoriness in that sense that a wider window of compositions can be used for fibers with a desired low shrinkage and high saline solubility.
The problem is solved by adding either or both of B_2O_3 and P_2O_5 in certain amounts to fiber compositions with certain amounts of SiO_2, CaO and MgO.
The addition of either or both of B_2O_3 and P_2O_5 to fiber compositions is known from D2 - D5.
However, it was not obvious to combine the teaching of D2 - D5 with D1, because
1. the addition of either or both of B_2O_3 and P_2O_5 in D2 - D5 was for a different reason (for improving saline solubility, and not for improving refractoriness), and
2. In D1 it is explicitly stated that impurities such as B_2O_3 are undesirable, if a certain resistance to temperature is to be achieved.
Therefore, inventive step is acknowledged.

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

The feature in claims 1, 5, 6 and 7 of the fibers "having a shrinkage of less than 3.5% when exposed to 800°C for 24 hours and having a shrinkage of less than 3.5% when exposed to 1000°C for 24 hours" is considered clear for the following reasons:
Said feature is not considered as a "result to be achieved", but as a product parameter which in itself is clear, distinctive and readily measurable.
The features mentioned on page 3, paragraphs 1 - 4, are not seen as restrictive, but as information enabling the skilled person to manufacture fibers that fulfill the shrinkage requirement.

SALINE SOLUBLE INORGANIC FIBRES

This invention relates to saline soluble inorganic fibres.

Saline soluble inorganic fibres have been described in several patent specifications, see for example WO93/15028. Fibres are required to be soluble in saline solution so that inhaled or ingested fibres dissolve rather than providing a source of irritation or otherwise affecting health. WO93/15028 showed that fibres comprising SiO_2, CaO and MgO and having a silica content of greater than 58% (or greater than 58% plus 0.5 times (wt%MgO - 10) if MgO > 10wt%) had suitable shrinkage characteristics at 800°C and 1000°C to be usable as refractory materials. A further feature of WO93/15028 was the use of the percentage of non-bridging oxygens present to predict the solubility of fibres in physiological saline solution.

Various subsequent applications have described the effect of P_2O_5 and B_2O_3 on solubility - see for example WO95/29135. P_2O_5 is alleged to have a solubilising effect on such fibres.

The German government have proposed a fibre classification which turns on a variable K_I which is defined as:

$$K_I = \Sigma(\text{Na,K,B,Ca,Mg,Ba -oxide}) - 2^* \text{ Al-oxide}$$

(the amounts of the oxides being expressed as weight %)

According to the proposed fibre classification if K_I is greater than 40 the fibre requires no health warnings. If K_I lies between 30 and 40 the fibre requires health warnings to be made. If K_I is less than 30 more serious marking is required (it is labelled as a carcinogen). It is readily apparent that it is difficult to provide a high K_I fibre (K_I>40) while still providing a refractory fibre like that of WO93/15028 (SiO_2>58wt%), there being a very narrow window of compositions to meet.

As a result of investigating fibre compositions that may meet the fibre classification and yet still be refractory enough to meet the standard of WO93/15028 (shrinkage of less than 3.5% at both 800°C and1000°C) the applicants have found that addition of P_2O_5 to compositions allows a broader range of refractory fibres to be produced than had previously been appreciated. They have also found that B_2O_3, previously thought to be

extremely detrimental to refractoriness, has a similar, although lesser, effect and that both P_2O_5 and B_2O_3 may be used in the fibres of WO93/15028.

The applicants have found that the refractoriness of the P_2O_5 and B_2O_3 containing fibres of the present invention is dependent on the sum of the amounts of SiO_2 and P_2O_5 (expressed in wt%)

It appears that a further factor that may be important in determining the refractoriness of a fibre is the percentage of non-bridging oxygens. If this percentage is 61.4% or more (calculated on the basis of the amounts of the components SiO_2, CaO, MgO, P_2O_5, and B_2O_3) the fibres tend to fail shrinkage tests at 800°C and 1000°C (failure being defined as a shrinkage of 3.5% or more).

Accordingly the present invention provides the use of P_2O_5 and/or B_2O_3 as a component to improve the refractoriness of inorganic fibres comprising SiO_2, and CaO and/or MgO, the inorganic fibres having a composition such that

$$SiO_2 + P_2O_5 - (58 + (\text{if } MgO > 10,\ 0.5 \times (MgO - 10) \text{ else } 0)) > -2.4\text{wt\%}$$

The invention provides further such fibres in which the percentage of non-bridging oxygens is less than 61.4%.

Further features of the invention are apparent from the claims in the light of the following description.

The percentage of non-bridging oxygens (%N.B.O.) is calculated by converting the weight percentages of SiO_2, CaO, MgO, P_2O_5, and B_2O_3 to molar amounts and inserting these amounts into the equation:-

$$\%N.B.O. = \frac{2*(CaO + MgO + P_2O_5 + B_2O_3)}{(2*SiO_2 + CaO + MgO + 5 \times P_2O_5 + 3 \times B_2O_3)} \times 100$$

The reason the amounts of CaO, MgO, P_2O_5, and B_2O_3 are doubled in the numerator to this equation is that each contributes two non-bridging oxygens. The reason terms are multiplied in the denominator to this equation is to reflect the number of oxygen atoms each molecular formula possesses.

Table I shows the results of a first set of shrinkage and solubility tests on compositions comprising SiO_2, CaO, MgO, P_2O_5, and B_2O_3 as main

CLAIMS

1. The use of P_2O_5 or B_2O_3 as a component to improve the refractoriness of inorganic fibres comprising SiO_2, and CaO and/or MgO, to produce inorganic fibres having a composition having a shrinkage of less than 3.5% when exposed to 1000°C for 24 hours and having a shrinkage of less than 3.5% when exposed to 800°C for 24 hours, the fibres having a composition such that

$$SiO_2 + P_2O_5 - (58 + (\text{if } MgO > 10,\ 0.5 \times (MgO - 10) \text{ else } 0)) > -2.4\text{wt\%}$$

2. The use of P_2O_5 or B_2O_3 as a component to improve the refractoriness of inorganic fibres as claimed in claim 1 in which the percentage of non-bridging oxygens is less than 61.4%.

3. The use of P_2O_5 or B_2O_3 as a component to improve the refractoriness of inorganic fibres as claimed in claim 1 or claim 2 in which the fibres fall within the compositional range:-

SiO_2	44 or more
CaO	20 - 40wt%
MgO	0 - 18wt%
P_2O_5	0- 12.5wt%
B_2O_3	0 - 4wt%

4. The use of P_2O_5 or B_2O_3 as a component to improve the refractoriness of inorganic fibres as claimed in claim 3 in which the fibres fall within the compositional range:-

SiO_2	52 - <58wt% [52 - <58+0.5'(MgO-10)wt% if MgO > 10wt%]
CaO	22 - 40wt%
MgO	0 - 17.5wt%
MgO + CaO	< 42wt%
P_2O_5	0.5 - 10wt%
B_2O_3	0 - 2wt%

5. The use of P_2O_5 or B_2O_3 as a component to improve the refractoriness of inorganic fibres as claimed in claim 3 in which the fibres fall within the compositional range:-

SiO_2	44.34 - 62.48

CaO	20.36 - 39.4wt%
MgO	0.62 - 21.16wt%
P_2O_5	0 - 12.01wt%
B_2O_3	0 - 3.54wt%

6. Saline soluble inorganic fibres having a shrinkage of less than 3.5% when exposed to 1000°C for 24 hours and having a shrinkage of less than 3.5% when exposed to 800°C for 24 hours, in which:-

$$SiO_2 + P_2O_5 - (58 + (\text{if } MgO > 10,\ 0.5 \times (MgO - 10) \text{ else } 0)) > -2.4wt\%$$

7. Saline soluble inorganic fibres as claimed in claim 6 comprising:-

SiO_2	44 or more
CaO	20 - 40wt%
MgO	0 - 18wt%
P_2O_5	0- 12.5wt%
B_2O_3	0 - 4wt%

8. Saline soluble inorganic fibres as claimed in claim 7 comprising:-

SiO_2	52 - <58wt% [52 - <58+0.5'(MgO-10)wt% if MgO > 10wt%]
CaO	22 - 40wt%
MgO	0 - 17.5wt%
MgO + CaO	< 42wt%
P_2O_5	0.5 - 10wt%
B_2O_3	0 - 2wt%

and in which the percentage of non-bridging oxygens calculated on the basis of the amounts of the above named components is less than 61.4%.

9. Saline soluble inorganic fibres as claimed in claim 7 comprising:-

SiO_2	44.34 - 62.48
CaO	20.36 - 39.4wt%
MgO	0.62 - 21.16wt%
P_2O_5	0 - 12.01wt%
B_2O_3	0 - 3.54wt%

10. Saline soluble inorganic fibres as claimed in claim 6 in which the fibres have a composition:-

SiO_2	52.4 - 57.85wt%
CaO	22.2 - 39.4wt%
MgO	1.96 - 17.4wt%
P_2O_5	0.82 - 7.8wt%
B_2O_3	0 - 1.95wt%
Al_2O_3	<1wt%